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(54) PROCESS FOR PREPARING 2,4- AND 2,6-TOLUYLENEDIAMINE

(71) We, CENTRALA INDUSTRIALA DE INGRASAMINTE CHIMICE, a Body Corporate duly organized under Rumanian Law, of Fagaras, Rumania, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a process for preparing 2,4- or 2,6-toluylenediamine by reduction of 2,4- or 2,6-dinitrotoluene.

Processes are known for preparing 2,4- and 2,6-toluylenediamines by reduction of dinitrotoluene with gases containing 50% of hydrogren, such as water gas, at a temperature of 70 to 150°C and pressure of from 5 to 500 atmospheres in the presence of copper-, cobalt- or nickel-containing catalysts.

These processes have the disadvantage that low yields, of up to 85%, are obtained.

The process according to the invention avoids these disadvantages in that the synthesis of 2,4- or 2,6-toluylenediamine is performed by reduction of 2,4- or 2,6-dinitrotoluene with ammonia synthesis gas, or purge gas obtained in the ammonia synthesis, in the presence of a Raney copper catalyst, at a temperature of about 150°C and pressure of about 50 atmospheres, using toluene as solvent. In this way yields substantially in excess of 90% are obtained.

Raney copper catalyst is a catalyst obtained by a process analagous to that used for the production of Raney nickel catalyst but substituting for the aluminium-nickel alloy used therein an aluminium-copper alloy.

The application of the invention is illustrated by the following three examples:

Example 1:

A 40% by weight solution containing a mixture of 80% by weight of dinitrotoluene and 20% by weight of recycled toluylenediamine with 2% by weight of Raney copper catalyst was introduced into a reaction vessel together with ammonia synthesis gas (H_2 75%, N_2 25% by volume) at 150°C and 50 atmospheres.

The excess of synthesis gas was recycled. Part of the crude reaction product was recycled and another part filtered and passed to a distillation column, where the toluene-water azeotrope was removed. The crude toluylenediamine was distilled in vacuo (10 mm Hg). The toluene, after separation from water, was recycled. The yield was 96% of toluylenediamine.

Example 2:

A solution having the same composition as that used in Example 1 was introduced together with 4% by weight of Raney copper catalyst into the reaction vessel and the operation was carried out under the same working conditions. After separation of the catalyst by filtration, the reaction product was processed by azeotropic distillation to remove water, then by vacuum distillation to obtain pure toluylenediamine. The yield was 98.8% of toluylenediamine.

Example 3:

A 40% solution containing a mixture of 80% dinitrotoluene and 20% recycled toluylenediamine dissolved in toluene, and 2% of Raney copper catalyst, was introduced into the reaction vessel together with a purge gas obtained in the synthesis of ammonia and having a composition of 60 volume % of H_2 and 40 volume % of N_2, at 50 atmospheres and 150°C.

The excess of reduction gas was recycled and so was 10 to 20% of the reaction product, the remainder being processed as described in Example 1. Yield: 97% of toluylenediamine.

The process according to the invention offers the advantage of higher yields and an important saving in costs by using the gas

used in the ammonia synthesis or purge gas obtained in the ammonia synthesis.

WHAT WE CLAIM IS:

1. A process for preparing 2,4- or 2,6-toluylenediamine by the catalytic reduction of 2,4- or 2,6-dinitrotoluene respectively in which the reaction is carried out in a toluene medium using ammonia sythesis gas, or purge gas obtained in the ammonia synthesis, at a temperature of about 150°C and a pressure of about 50 atmospheres and in the presence of a Raney copper catalyst.

2. A process according to Claim 1 substantially as described in any of the Examples herein.

3. 2,4- or 2,6-toluylenediamine whenever prepared by a process according to Claim 1 or Claim 2.

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